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Solar physical vapor deposition: A new approach for preparing magnesium titanate nanopowders

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ABSTRACT

Solar energy is a major factor in the equation of energy, because of the unlimited potential of the sun that eclipses all other renewable sources of energy. Solar physical vapor deposition (SPVD) is a core innovative, original and environmentally friendly process to prepare nanocrystalline materials in a powder form. The principle of this process is to melt the material under concentrated solar radiation, which evaporates and condenses as nanopowders on a cold surface. We synthesized nanopowders of magnesium titanate by the SPVD process at PROMES Laboratory in Odeillo-Font Romeu, France. The SPVD system consists of a parabolic mirror concentrator, a mobile plane mirror ("heliostat") tracking the sun and a solar reactor "heliotron". The synthesized nanopowders were analyzed by X-ray diffraction (XRD) to know their crystalline structure and scanning electron microscopy (SEM) was used for determining the surface morphology. We have shown that the characteristics of obtained nanotitanates were determined by the targets' composition and SPVD process parameters such as the working pressure inside the solar reactor and evaporation duration (process time).

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1. Introduction

To guarantee uninterrupted energy supplies for the future it is not only necessary to continue developing traditional technologies but also to promote new processes. Renewable energy sources essentially have a major role in decreasing emissions of greenhouse gases and preventing climate changes.

The sun is the most abundant source of energy on the planet. The long-term fossil fuel reserve will deplete in the years to come and probably solar power will become the most dominant energy source to the world. The development of clean and inexhaustible solar energy technologies will have high benefits and advantages such as: control pollution, global warming, keep petroleum prices under check and increase energy independence of the countries. The applications of solar energy is expected to meet the demands of industrial and domestic needs including: (a) electricity generation by conversion of sunlight into electricity directly by photovoltaics (photoelectric effect) or indirectly by concentrators (mirrors or lenses and tracking systems), (b) production of hydrogen, (c) water and space heating, (d) water treatment by solar distillation, (e) solar cookers for cooking, drying and pasteurization, (f) day lighting systems for interior illumination, (g) agriculture and horticulture to optimize the productivity of plants, heating greenhouses, and (h) research in advanced materials.

Nanomaterials are of particular interest due to their improved chemical, electrical, optical, magnetic and mechanical properties when compared to their bulk counterpart. The change in properties is attributed to their large surface area to volume ratio, which provides huge forces for diffusion, sintering at lower temperature over shorter durations and lower melting temperature. The improved properties of nanomaterials compared to the conventional bulk materials indicate that their exploration will be scientifically interesting and technologically important. Due to these trends, nowadays, research on nanomaterials has become increasingly important for application in nano/microelectronics, spintronics, optoelectronics, chemical (novel reagents, catalysts), automotive, aerospace, construction industries, health care (implants, nanomedicine, tissue engineering, etc.) and agriculture. In addition, microelectronics industry continues to scale down device size. Small size, light weight, optimum performance and low power consumption are the key aspects for the current and next generation of microelectronics.

Growing interest in nanostructured materials has led to a variety of new methodologies for manufacturing novel materials with the possibility of engineering their properties in the nano scale. Nanomaterials are being prepared by various techniques such as,

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Table 1
SPVD parameters for $(MgO)_{0.40}(TiO_2)_{0.41}$ [sample code: $MgTiO_{3IPEE}$].

SPVD parameters	Sample code: $MgTiO_{3IPEE}$ powder	Sample code: $MgTiO_{3IPEE}$ pressed disc 10 mm diameter × 5–6 mm thick
Sun flux (W/m^2)	786–845	875–915
Holder height (mm)	60.4	60.4
Sample height (ΔH, mm)	4–5	5–6
Ultimate pressure (hPa)	120	120
Working pressure (hPa)	30	120
Atmosphere	Air	Air
Duration (min)	270	360
Obtained sample code	VAiFMgTIPEE-2 – sample from Filter VAiCFMgTIPEE-2 – sample from cold finger VAiMgTIPEEres-2 – residue from target	VAiFMgTIPEE-1 – sample from Filter VAiCFMgTIPEE-1 – sample from cold finger VAiMgTIPEEres-1 – residue from target

chemical or physical vapor deposition (CVD or PVD) [1,2], sol–gel [3], hydrothermal [4], precipitation methods [5]. Also, nanostructured thin films can be obtained by pulsed laser deposition (PLD) [6,7], radio frequency magnetron sputtering (RFMS) [8], laser molecular beam epitaxy (MBE) [9] and microwave flash synthesis [10]. Dense nanomaterials can be obtained by fast sintering or spark plasma sintering (SPS).

Concentrated solar power technology has successfully demonstrated its capability of converting solar radiation into high-temperature heat. The produced power is the cleanest and most efficient of renewable energy. As a result, an original process to prepare nanopowders was successfully developed as solar physical vapor deposition at CNRS-PROMES Laboratory in Odeillo-Font Romeu, France. SPVD is a process in which a material is melted and vaporized (or sublimated) and condensed in a solar reactor using concentrated solar radiation. The SPVD process has been employed to prepare various kinds of nanomaterials that encompass but not restricted to pure ZnO or doped $Zn_{1-x}Al_xO$, $Zn_{1-x}Bi_xO$, $Zn_{1-x}Co_xO$, $Zn_{1-x}Sb_xO$, $Zn_{1-x}In_xO$, Zn and Al metallic [11–16], TiO_2 pure or doped with Fe, Co or Mn [17], Si and SiO_x [18], maghemite γ-Fe_2O_3 [19], β-$Bi_2O_{3-\delta}$, In_2O_3, SnO_2 [20], yttria doped zirconia $Zr_{1-x}Y_xO_{2-\delta}$ [21], gadolinium doped ceria $Ce_{1-x}Ga_xO_{2-\delta}$ and also fullerenes [22] and carbon nanotubes [23]. Some potential applications of these nanophases are: $H_2(g)$ production by reduction of water, solar energy storage, luminescent screens, sensors, in optoelectronics, transparent electrodes, electrolytes in solid oxide fuel cells (SOFC), varistors, quantum dots, spintronics and magnetic traces in medicine.

The next generation of functional ceramics requires tailoring materials to meet the specific needs of the application. For which, it is necessary to investigate how the process parameters, microstructure/nanostructure and composition variation influence the properties.

Usually, in chemistry, titanate refers to inorganic compounds composed of titanium oxides. The metatitanates have a general formula $MTiO_3$ where M is a divalent element. Among many kinds of known titanates, magnesium titanate is considered to be commercially important due to their superior dielectric properties. Magnesium titanate with the chemical formula $MgTiO_3$ has many industrial applications such as, high frequency capacitors, temperature compensating capacitors and chip capacitors [24,25]. Microwave dielectrics having high Q (quality factor, $Q = 1/\tan\delta$) values at a frequency above 10 GHz are used in satellite communication and broadcasting systems [26]. Thus, $MgTiO_3$ was selected as a model material for the present study. It is well known that the dielectric properties of these materials have a strong dependence on their microstructure, a property that can be controlled by choosing the appropriate synthesis technique and processing conditions. $MgTiO_3$ (geikielite) has a rhombohedral structure (space group R^{-3}) with $a = 5.05$ Å and $c = 13.89$ Å and it is the magnesium analogue of ilmenite.

2. Materials and methods

2.1. Targets preparation

Magnesium titanate powders with the general formula $(MgO)_x(TiO_2)_y$, where x/y is the molar ratio, were prepared by the conventional solid-state reaction method employing MgO (Rhone Poulenc, France, >99%) and TiO_2 (PreTiO_2X, Czech Republic, >99%). In the present study, the x/y molar ratio was varied and two compositions, $(MgO)_{0.40}(TiO_2)_{0.41}$ and $(MgO)_{0.52}(TiO_2)_{0.30}$, were obtained. The desired amounts of the starting materials were thoroughly mixed for 2 h in ethanol using a ball milling with agate balls and jars. The resulting powders were subsequently dried at 120 °C, sieved and then calcined at 1000 °C for 1 h, in air, in an electric chamber furnace. To ensure good homogeneity, the calcined powders were re-milled under similar conditions.

The synthesis of these materials (used further as targets in SPVD process) via conventional solid-state reaction ceramic route was done at S.C. IPEE ATI S.A. Curtea de Arges, Romania.

Table 2
SPVD parameters for $(MgO)_{0.52}(TiO_2)_{0.30}$ [sample code: SB5].

SPVD parameters	Sample code: SB5 powder	Sample code: SB5 pressed disc 10 mm diameter × 5–6 mm thick
Sun flux (W/m^2)	824–845	870–880
Holder height (mm)	60.1	60.8
Sample height (ΔH, mm)	3	5–6
Ultimate pressure (hPa)	120	120
Working pressure (hPa)	90	120
Atmosphere	Air	Air
Duration (min)	70	210
Obtained sample code	VAiFSB5-1 – sample from filter VAiSB5res-1 – residue from target	VAiFSB5-2 – sample from filter VAiCFSB5-2 – sample from cold finger VAiSB5res-2 – residue from target

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